

Claims

What is claimed is:

- 5           1.     A method of manufacturing an optical waveguide preform, said method comprising:
- providing a first doping atmosphere to a soot preform contained in a vessel;
- holding the first doping atmosphere in the vessel for a first reacting time
- 10           sufficient to at least partially dope the soot preform;
- at least partially refilling the vessel with a second doping atmosphere;
- and
- holding the second doping atmosphere in the vessel for a second reacting time sufficient to further dope the soot preform.
- 15           2.     The method of Claim 1 further including, following said step of holding the second doping atmosphere:
- at least partially refilling the vessel with a third doping atmosphere; and
- holding the third doping atmosphere in the vessel for a third reacting
- 20           time sufficient to further dope the soot preform.
3.     The method of Claim 1 including:
- pressurizing the first doping atmosphere about the soot preform during the first reacting time;
- 25           depressurizing the first doping atmosphere about the soot preform at the end of the first reacting time; and
- pressurizing the second doping atmosphere about the soot preform during the second reacting time.
- 30           4.     The method of Claim 1 including increasing the pressure of at least one of the first doping atmosphere during the first reacting time or the second doping atmosphere during the second reacting time.

5. The method of Claim 4 wherein the vessel is substantially completely gas-sealed throughout said step of increasing the pressure.

6. The method of Claim 1 wherein:

5 each of the first and second doping atmospheres includes a dopant gas; and

from the beginning of the first reacting time to the end of the second reacting time, the total pressure of the atmosphere in vessel is maintained substantially constant and the partial pressure of the dopant gas is varied.

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7. The method of Claim 6 wherein an inert gas is added to the atmosphere in the vessel during and between said steps of holding the first doping atmosphere and holding the second doping atmosphere to compensate for reductions in the partial pressure of the dopant gas resulting from reaction of the dopant gas with the soot preform.

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8. The method of Claim 6 wherein the dopant gas is added to the atmosphere in the vessel during at least one of the first and second reacting times to compensate for reductions in the partial pressure of the dopant gas resulting from reaction of the dopant gas with the soot preform.

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9. The method of Claim 6 further including the step of at least partially purging any remaining doping atmosphere and any products of reaction from the vessel prior to said step of refilling the vessel with the second doping atmosphere.

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10. The method of Claim 1 wherein:

each of the first and second doping atmospheres includes a dopant gas; and

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the dopant gas is added to the atmosphere in the vessel during at least one of the first and second reacting times to compensate for reductions in the partial pressure of the dopant gas resulting from reaction of the dopant gas with the soot preform.

11. The method of Claim 1 including pressurizing an outer surface of the vessel to offset pressurization within the vessel.

5 12. The method of Claim 1 including supporting a reinforcing sleeve about the vessel during at least the first and second reacting times.

13. The method of Claim 1 including rotating the soot preform relative to the vessel and wherein the vessel is sealed.

10 14. The method of Claim 1 including:  
drying the soot preform prior to said step of providing the first doping atmosphere; and  
sintering the soot preform following the second reacting time.

15 15. The method of Claim 14 wherein the soot preform remains in the pressure chamber throughout each of the steps recited in Claims 1 and 14.

20 16. The method of Claim 1 including:  
pressurizing the first doping atmosphere to a gage pressure of at least 0.1 atm gage during the first reacting time; and  
pressurizing the second doping atmosphere to a gage pressure of at least 0.1 atm gage during the second reacting time.

25 17. The method of Claim 1 including:  
pressurizing the first doping atmosphere to a first pressure during the first reacting time; and  
pressurizing the second doping atmosphere to a second pressure during the second reacting time;  
wherein the second pressure is different than the first pressure.

30 18. The method of Claim 1 including increasing a total pressure of the doping atmosphere in the vessel during the reacting time.

19. The method of Claim 1 wherein the first reacting time is between about 1 and 60 minutes.

5 20. The method of Claim 1 wherein the second reacting time is between about 1 and 60 minutes.

21. The method of Claim 1 wherein the second reacting time is longer than the first reacting time.

10 22. The method of Claim 1 including:  
heating the first doping atmosphere to a temperature of between about 200 and 1350 °C during the first reacting time; and  
heating the second doping atmosphere to a temperature of between about 200 and 1350 °C during the second reacting time.

15 23. The method of Claim 1 including:  
heating the first doping atmosphere to a first temperature during the first reacting time; and  
heating the second doping atmosphere to a second temperature during  
20 the second reacting time;  
wherein the second temperature is different from the first temperature.

24. The method of Claim 1 wherein the first doping atmosphere is simultaneously maintained in a quartz muffle and at a temperature less than 1200 C  
25 during the first reacting time.

25. The method of Claim 1 wherein each of the first and second doping atmospheres includes a halogen-containing compound.

30 26. The method of Claim 25 wherein the first and second halogen-containing compounds are the same.

27. The method of Claim 25 wherein the first and second doping atmospheres each include a halogen-containing compound selected from the group consisting of  $\text{SiF}_4$ ,  $\text{SF}_6$ ,  $\text{CF}_4$ ,  $\text{C}_2\text{F}_6$ ,  $\text{COF}_2$ ,  $\text{C}_2\text{F}_2\text{Cl}_2$ ,  $\text{F}_2$ , and  $\text{Cl}_2$ .

5 28. The method of Claim 25 wherein the first and second doping atmospheres each include a fluorine-containing compound.

29. The method of Claim 1 wherein the first and second doping atmospheres each include an inert gas selected from the group consisting of He, Ar, Ne, and  $\text{N}_2$ .

10 30. A method of manufacturing an optical waveguide preform, said method comprising:

providing a doping atmosphere to a soot preform contained in a vessel;

holding the doping atmosphere in the vessel for a reacting time of

15 between about 1 and 60 minutes to at least partially dope the soot preform; and

evacuating at least a portion of the doping atmosphere from the vessel at the end of the reacting time.

31. The method of Claim 30 including:

20 at least partially refilling the vessel with a second doping atmosphere following the reacting time; and

holding the second doping atmosphere in the vessel for a second reacting time sufficient to further dope the soot preform.

25 32. The method of Claim 30 including:

pressurizing the doping atmosphere about the soot preform for the reacting time; and

depressurizing the doping atmosphere about the soot preform at the end of the reacting time.

30 33. The method of Claim 32 wherein the vessel is substantially completely gas-sealed throughout said step of pressurizing the doping atmosphere.

34. The method of Claim 32 further including, following said step of depressurizing the doping atmosphere:

replacing at least a portion of the doping atmosphere with a second doping atmosphere about the soot preform; and

5           pressurizing the second doping atmosphere about the soot preform for a second reacting time of no more than 60 minutes to further dope the soot preform.

10           35. The method of Claim 34 wherein said step of pressurizing the second doping atmosphere includes pressurizing the second doping atmosphere to a gage pressure of at least 0.1 atm.

15           36. The method of Claim 35 wherein said step of pressurizing the second doping atmosphere includes pressurizing the second doping atmosphere to a gage pressure of at least 0.5 atm.

37. The method of Claim 34 wherein the second reacting time is between about 5 and 30 minutes.

20           38. The method of Claim 34 wherein said step of pressurizing the second doping atmosphere includes heating the second doping atmosphere to a temperature of between about 1125 and 1300 °C.

25           39. The method of Claim 30 including pressurizing an outer surface of the vessel to offset pressurization within the vessel.

40. The method of Claim 30 including rotating the soot preform relative to the vessel.

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41. The method of Claim 30 including:  
drying the soot preform prior to said step of providing the doping atmosphere; and  
sintering the soot preform following the reacting time.

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42. The method of Claim 30 including pressurizing and heating the doping atmosphere about the soot preform to a gage pressure of at least 0.1 atm and a temperature of less than 1350 °C.

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43. The method of Claim 30 including pressurizing the doping atmosphere to a gage pressure of at least 0.1 atm gage.

44. The method of Claim 43 including pressurizing the doping atmosphere to a gage pressure of at least 0.5 atm gage.

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45. The method of Claim 30 wherein the reacting time is between about 5 and 30 minutes.

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46. The method of Claim 30 including heating the doping atmosphere to a temperature of between about 1000 and 1350 °C.

47. The method of Claim 30 wherein the doping atmosphere includes a halogen-containing compound selected from the group consisting of SiF<sub>4</sub>, SF<sub>6</sub>, CF<sub>4</sub>, C<sub>2</sub>F<sub>6</sub>, COF<sub>2</sub>, C<sub>2</sub>F<sub>2</sub>Cl<sub>2</sub>, F<sub>2</sub>, and Cl<sub>2</sub>.

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48. The method of Claim 30 wherein the doping atmosphere includes a fluorine-containing compound.

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49. The method of Claim 48 wherein the doping atmosphere includes a fluorine-containing compound selected from the group consisting of SiF<sub>4</sub>, SF<sub>6</sub>, CF<sub>4</sub>, C<sub>2</sub>F<sub>6</sub>, COF<sub>2</sub>, C<sub>2</sub>F<sub>2</sub>Cl<sub>2</sub>, and F<sub>2</sub>.

50. The method of Claim 49 wherein the doping atmosphere includes a fluorine-containing compound selected from the group consisting of  $\text{SiF}_4$ ,  $\text{SF}_6$ , and  $\text{CF}_4$ .

5 51. A method of manufacturing an optical waveguide preform, said method comprising:

flowing a pulse of a process gas into a vessel to form a doping atmosphere in the vessel about a soot preform, the process gas including a doping gas;

10 maintaining the soot preform in contact with the doping atmosphere for a reacting time sufficient to at least partially dope the soot preform; and

evacuating at least a portion of the doping atmosphere from the vessel at the end of the reacting time.

15 52. The method of claim 51 further including flowing a makeup gas into the vessel during the reacting time, wherein the flow rate of the makeup gas is less the flow rate of the pulse of the process gas.

20 53. The method of claim 52 wherein the flow rate of the makeup gas is provided to at least partially offset for any pressure loss due to the doping gas reacting with the preform.

25 54. The method of claim 51 wherein the doping atmosphere is maintained at ambient pressure about the soot preform during the reacting time.

55. The method of claim 51 wherein the doping atmosphere is pressurized about the soot preform to a doping pressure greater than ambient pressure during the reacting time.

30 56. The method of claim 51 including flowing a second pulse of the process gas into the vessel to form a second doping atmosphere in the vessel about a soot preform following said step of evacuating at least a portion of the doping atmosphere from the vessel.



57. A method of manufacturing an optical waveguide preform, said method comprising:

- 5       providing a first drying atmosphere to a soot preform contained in a vessel;
- holding the first drying atmosphere in the vessel for a first reacting time sufficient to at least partially dry the soot preform;
- at least partially refilling the vessel with a second drying atmosphere; and
- 10       holding the second drying atmosphere in the vessel for a second reacting time sufficient to further dry the soot preform.

58. The method of Claim 57 further including, following said step of holding the second drying atmosphere:

- 15       at least partially refilling the vessel with a third drying atmosphere; and
- holding the third drying atmosphere in the vessel for a third reacting time sufficient to further dry the soot preform.

59. The method of Claim 57 including:

- 20       pressurizing the first drying atmosphere about the soot preform during the first reacting time;
- depressurizing the first drying atmosphere about the soot preform at the end of the first reacting time; and
- pressurizing the second drying atmosphere about the soot preform
- 25       during the second reacting time.

60. The method of Claim 57 including increasing the pressure of at least one of the first drying atmosphere during the first reacting time or the second drying atmosphere during the second reacting time.

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61. The method of Claim 60 wherein the vessel is substantially completely gas-sealed throughout said step of increasing the pressure.

62. The method of Claim 57 wherein:

each of the first and second drying atmospheres includes a drying gas;

from the beginning of the first reacting time to the end of the second

reacting time, the total pressure of the atmosphere in vessel is maintained

substantially constant and the partial pressure of the drying gas is varied.

63. The method of Claim 62 wherein an inert gas is added to the atmosphere

in the vessel during and between said steps of holding the first drying atmosphere and

holding the second drying atmosphere to compensate for reductions in the partial

pressure of the drying gas resulting from diffusion of the drying gas into the soot

preform.

64. The method of Claim 62 wherein the drying gas is added to the

atmosphere in the vessel during at least one of the first and second reacting times to

compensate for reductions in the partial pressure of the drying gas resulting from

diffusion of the drying gas into the soot preform.

65. The method of Claim 62 further including the step of purging the drying

atmosphere from the vessel prior to said step of refilling the vessel with the second

drying atmosphere.

66. The method of Claim 57 wherein:

each of the first and second drying atmospheres includes a drying gas;

and

the drying gas is added to the atmosphere in the vessel during at least

one of the first and second reacting times to compensate for reductions in the

partial pressure of the drying gas resulting from diffusion of the drying gas into

the soot preform.

67. The method of Claim 57 including pressurizing an outer surface of the

vessel to offset pressurization within the vessel.

68. The method of Claim 57 including supporting a reinforcing sleeve about the vessel during at least the first and second reacting times.

69. The method of Claim 57 including rotating the soot preform relative to the vessel.

70. The method of Claim 57 including sintering the soot preform following the second reacting time.

71. The method of Claim 70 wherein the soot preform remains in the pressure chamber throughout each of the steps recited in Claims 1 and 70.

72. The method of Claim 57 including:  
pressurizing the first drying atmosphere to a gage pressure of at least 0.1 atm gageduring the first reacting time; and  
pressurizing the second drying atmosphere to a gage pressure of at least 0.1 atm gageduring the second reacting time.

73. The method of Claim 57 including:  
pressurizing the first drying atmosphere to a first pressure during the first reacting time; and  
pressurizing the second drying atmosphere to a second pressure during the second reacting time;  
wherein the second pressure is different than the first pressure.

74. The method of Claim 57 including increasing a total pressure of the drying atmosphere in the vessel during the reacting time.

75. The method of Claim 57 wherein the first reacting time is between about 1 and 60 minutes.

76. The method of Claim 57 wherein the second reacting time is between about 5 and 30 minutes.

77. The method of Claim 57 wherein the second reacting time is longer than the first reacting time.

5           78. The method of Claim 57 including:  
              heating the first drying atmosphere to a temperature of between about  
              300 and 1200 °C during the first reacting time; and  
              heating the second drying atmosphere to a temperature of between about  
              300 and 1200 °C during the second reacting time.

10           79. The method of Claim 57 including:  
              heating the first drying atmosphere to a first temperature during the first  
              reacting time; and  
              heating the second drying atmosphere to a second temperature during  
15           the second reacting time;  
              wherein the second temperature is different from the first temperature.

20           80. The method of Claim 57 wherein the first drying atmosphere is  
              simultaneously maintained in a quartz muffle and at a temperature less than 1200 °C  
              during the first reacting time.

              81. The method of Claim 57 wherein each of the first and second drying  
              atmospheres includes a halogen-containing compound.

25           82. The method of Claim 81 wherein the first and second halogen-containing  
              compounds are the same.

30           83. The method of Claim 81 wherein the first and second drying  
              atmospheres each include a halogen-containing compound selected from the group  
              consisting of Cl<sub>2</sub>, SiCl<sub>4</sub>, GeCl<sub>4</sub>, SOCl<sub>2</sub>, or POCl<sub>3</sub>.

84. The method of Claim 57 wherein the first and second drying atmospheres each include an inert gas selected from the group consisting of He, Ar, Ne, and N<sub>2</sub>.

5           85. A method of manufacturing an optical waveguide preform, said method comprising:

                  providing a drying atmosphere to a soot preform contained in a vessel;  
                  holding the drying atmosphere in the vessel for a reacting time of  
                  between about 1 and 60 minutes to at least partially dry the soot preform; and  
10                evacuating at least a portion of the drying atmosphere from the vessel at  
                  the end of the reacting time.

                  86. The method of Claim 85 including:  
                  at least partially refilling the vessel with a second drying atmosphere  
15                following the reacting time; and  
                  holding the second drying atmosphere in the vessel for a second reacting  
                  time sufficient to further dry the soot preform.

                  87. The method of Claim 85 including:  
20                pressurizing the drying atmosphere about the soot preform for the  
                  reacting time; and  
                  depressurizing the drying atmosphere about the soot preform at the end  
                  of the reacting time.

25           88. The method of Claim 87 wherein the vessel is substantially completely  
gas-sealed throughout said step of pressurizing the drying atmosphere.

                  89. The method of Claim 87 further including, following said step of  
depressurizing the drying atmosphere:  
30                replacing at least a portion of the drying atmosphere with a second  
                  drying atmosphere about the soot preform; and

pressurizing the second drying atmosphere about the soot preform for a second reacting time of no more than 30 minutes to further dry the soot preform.

5           90.    The method of Claim 89 wherein said step of pressurizing the second drying atmosphere includes pressurizing the second drying atmosphere to a gage pressure of at least 0.1 atm gage.

10           91.    The method of Claim 90 wherein said step of pressurizing the second drying atmosphere includes pressurizing the second drying atmosphere to a gage pressure of at least 0.5 atm gage.

15           92.    The method of Claim 89 wherein the second reacting time is between about 5 and 30 minutes.

          93.    The method of Claim 89 wherein said step of pressurizing the second drying atmosphere includes heating the second drying atmosphere to a temperature of between about 850 and 1100 °C.

20           94.    The method of Claim 85 including pressurizing an outer surface of the vessel to offset pressurization within the vessel.

          95.    The method of Claim 85 including rotating the soot preform relative to the vessel.

25           96.    The method of Claim 85 including sintering the soot preform following the reacting time.

30           97.    The method of Claim 85 including pressurizing and heating the drying atmosphere about the soot preform to a gage pressure of at least 0.1 atm and a temperature of less than 1200 °C.

98. The method of Claim 85 including pressurizing the drying atmosphere to a gage pressure of at least 0.1 atm.

5 99. The method of Claim 98 including pressurizing the drying atmosphere to a gage pressure of at least 0.5 atm.

100. The method of Claim 85 wherein the reacting time is between about 5 and 30 minutes.

10 101. The method of Claim 85 including heating the drying atmosphere to a temperature of between about 850 and 1100 °C.

15 102. The method of Claim 85 wherein the drying atmosphere includes a halogen-containing compound selected from the group consisting of Cl<sub>2</sub>, SiCl<sub>4</sub>, GeCl<sub>4</sub>, SOCl<sub>2</sub>, or POCl<sub>3</sub>.

20 103. A method of manufacturing an optical waveguide preform, said method comprising:  
    flowing a pulse of a process gas into a vessel to form a drying atmosphere in the vessel about a soot preform, the process gas including a drying gas;  
    maintaining the soot preform in contact with the drying atmosphere for a reacting time sufficient to at least partially dry the soot preform; and  
    evacuating at least a portion of the drying atmosphere from the vessel at  
25 the end of the reacting time.

104. The method of claim 103 wherein the drying atmosphere is maintained at ambient pressure about the soot preform during the reacting time.

30 105. The method of claim 103 wherein the drying atmosphere is pressurized about the soot preform to a drying pressure greater than ambient pressure during the reacting time.

106. The method of claim 103 including flowing a second pulse of the process gas into the vessel to form a second drying atmosphere in the vessel about a soot preform following said step of evacuating at least a portion of the drying atmosphere from the vessel.

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107. An apparatus for manufacturing an optical waveguide preform using a soot preform, said apparatus comprising:

a pressure vessel defining a pressure chamber adapted to contain the soot preform;

10 a supply of process gas including at least one of a doping gas and a drying gas;

a fluid control system including:

a flow control device operable to prevent and allow flow of said process gas into and out of said pressure chamber to form an atmosphere about the soot preform; and

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a pressurizing device operable to pressurize said atmosphere in said pressure chamber;

a process controller operative to control said flow control device and said pressurizing device to:

20 pressurize said atmosphere about the soot preform to a selected pressure for a reacting time of between about 1 and 30 minutes to dope or dry the soot preform; and

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depressurize said atmosphere about the soot preform at the end of said reacting time; and

25 a heater operable to heat said atmosphere and the preform in said pressure chamber.

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108. The apparatus of Claim 107 wherein said process controller is operative to pressurize said atmosphere about the soot preform to a gage pressure of at least 0.1 atm gage.

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109. The apparatus of Claim 107 wherein said process gas includes a halogen-containing compound.



110. The apparatus of Claim 109 wherein said process gas includes a halogen-containing compound selected from the group consisting of  $\text{Cl}_2$ ,  $\text{GeCl}_4$ ,  $\text{SiCl}_4$ ,  $\text{SiF}_4$ ,  $\text{SF}_6$ ,  $\text{CF}_4$ ,  $\text{C}_2\text{F}_6$ ,  $\text{COF}_2$ ,  $\text{C}_2\text{F}_2\text{Cl}_2$ , and  $\text{F}_2$ .

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111. The apparatus of Claim 107 wherein said process gas includes a fluorine-containing compound.

112. The apparatus of Claim 111 wherein said process gas includes a fluorine-containing compound selected from the group consisting of  $\text{SiF}_4$ ,  $\text{SF}_6$ ,  $\text{CF}_4$ ,  $\text{C}_2\text{F}_6$ ,  $\text{COF}_2$ ,  $\text{C}_2\text{F}_2\text{Cl}_2$ , and  $\text{F}_2$ .

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113. The apparatus of Claim 112 wherein said process gas includes a fluorine-containing compound selected from the group consisting of  $\text{SiF}_4$ ,  $\text{SF}_6$ , and  $\text{CF}_4$ .

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114. The apparatus of Claim 107 wherein said process gas supply includes a mixture of said at least one of a dopant gas and a drying gas and an inert gas.

115. The apparatus of Claim 107 including a supply of inert gas and wherein said fluid control system is operable to prevent and allow flow of said inert gas into and out of said pressure chamber to form a portion of said atmosphere about the soot preform.

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116. The apparatus of Claim 107 wherein said flow control device includes at least one valve.

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117. The apparatus of Claim 116 wherein said at least one valve includes a mass flow controller.

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118. The apparatus of Claim 107 wherein said pressurizing device includes at least one of:

a compressor; and  
a pressurized tank and a regulator.

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119. The apparatus of Claim 107 including a holding assembly operative to hold and rotate the soot preform in said pressure chamber.

120. The apparatus of Claim 119 wherein said holding assembly includes a holder adapted to directly engage and support the soot preform and a drive unit non-mechanically coupled to said holder and operable to rotate said holder relative to said chamber.

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121. The apparatus of Claim 120 wherein said drive unit is magnetically coupled to said holder.

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122. The apparatus of Claim 107 including a reinforcing sleeve surrounding at least a portion of said pressure vessel.

123. The apparatus of Claim 122 wherein said reinforcing sleeve is formed of a material selected from the group consisting of silicon carbide, silicon nitride, graphite, and alumina.

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124. The apparatus of Claim 107 including a second pressure vessel surrounding at least a portion of said first pressure vessel, said first and second pressure vessels defining a second pressure chamber therebetween.

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125. The apparatus of Claim 107 wherein said wherein substantially all of the portions of said pressure vessel interfacing with said pressure chamber are formed of quartz.

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126. The apparatus of Claim 107 including a second heater.

127. The apparatus of Claim 107 wherein said heater is a variable temperature heater.

5 128. An apparatus for manufacturing an optical waveguide preform using a soot preform and an atmosphere, said apparatus comprising:

- a) a first pressure vessel having inner and outer opposed surfaces, said inner surface defining a first pressure chamber adapted to contain the soot preform and the atmosphere;
- 10 b) a second pressure vessel surrounding at least a portion of said first pressure vessel, said first and second pressure vessels defining a second pressure chamber therebetween; and
- c) a pressurizing system operable to pressurize each of the first and second pressure chambers to control a differential between pressures on said inner and outer surfaces of said first pressure vessel.

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129. The apparatus of Claim 128 wherein substantially all of said inner surface of said first pressure vessel that interfaces with said first pressure chamber is formed of quartz.

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130. The apparatus of Claim 128 wherein said pressurizing system includes a compressor operable to pressurize said first chamber.

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131. The apparatus of Claim 128 adapted to allow said first pressure vessel to be pressurized to a gage pressure of at least 0.5 atm and heated to a temperature of at least 1000 °C without displacing said inner surface more than 20%.